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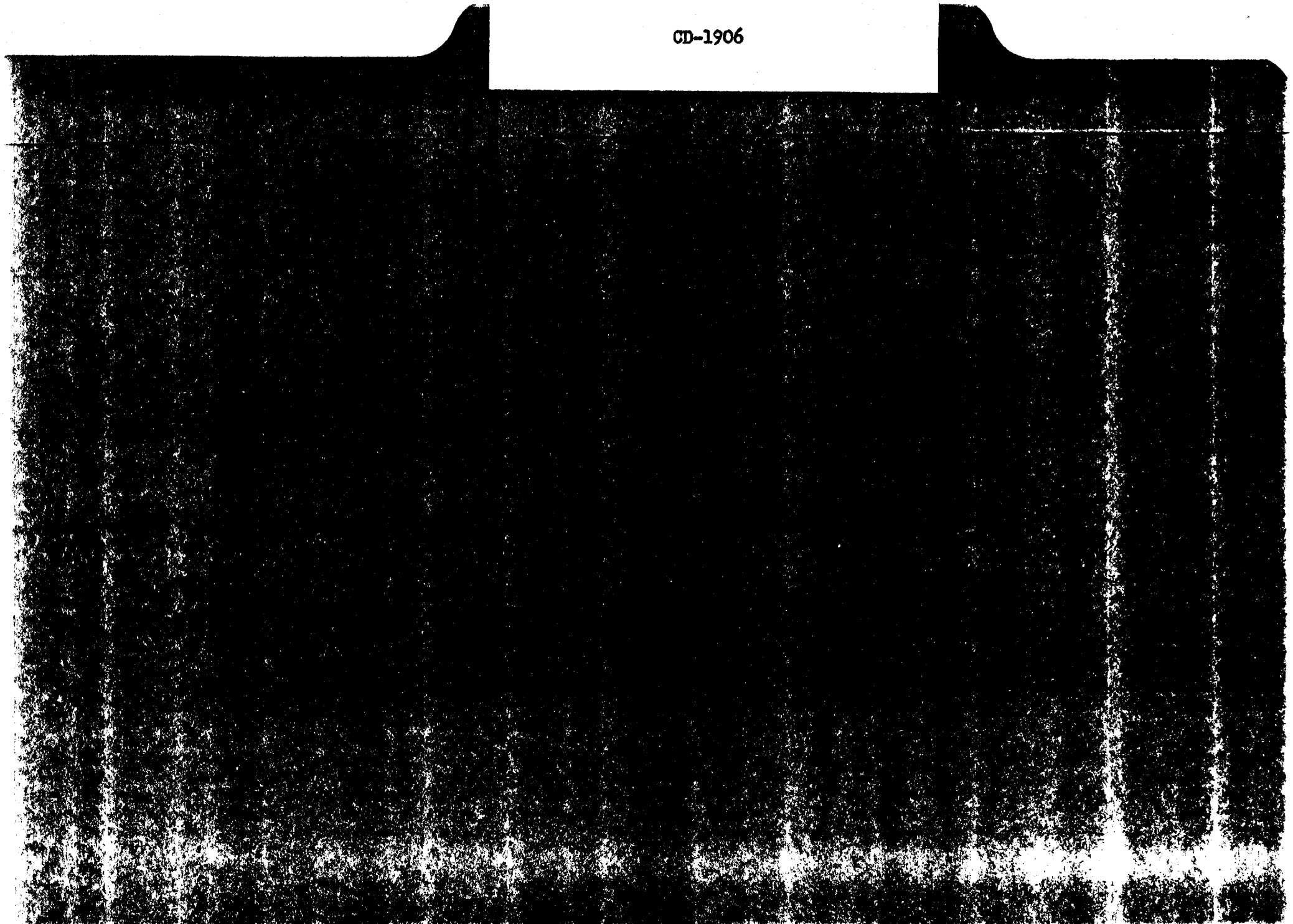

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A SURVEY OF SAMPLING METHODS AND SAMPLES

I. INTRODUCTION

A. The Problem

This survey was initiated because of widespread dissatisfaction with many of the samples presented to the Analytical Laboratories for analysis.

It was also thought that some analyses, originally essential in experimental stages of the process, might prove on critical inspection to have out-lived their usefulness.

B. Limitations of the Survey

Experimental samples which are not taken consistently have been omitted.

A few analyses are regarded as satisfactory, in spite of a large tolerance of error, because they are only approximate checks to govern certain steps in the process. Such analyses are usually accompanied by more precise analyses on duplicate samples, for the purpose of keeping the material balance.

C. Current Efforts to Improve Sampling

An inspector for sampling has been designated (Mr. Warnock), who has prepared procedures to be followed in taking samples. These procedures have been approved by Mr. Thornton, and their application in practice has been observed by Mr. Warnock and described in his reports.

Serious consideration is now being given to the establishment of a sampling laboratory, to handle material which cannot be readily processed by the source department.

II. DIFFICULTIES IN SAMPLING

Observation of the sampling procedures as they are carried out at present shows two apparent sources of difficulty, which frequently result in non-representative samples and lack of precision in analyses.

If non-homogeneous material is produced during track or chemical operation, such material requires thorough mixing, or if a solid, grinding and mixing, before a representative sample can be taken. If even a moderate quantity of such material is produced, mechanical mixing becomes necessary, which is at present beyond the facilities now available, either in the source departments or the laboratories. Even the Salvage Department may not be able to take the considerable time necessary to process individual batches of material from a series of experimental runs, although analyses of these individual batches may be necessary for adjustment and operation. The only recourse then, may be a hastily taken and non-representative sample, resulting in discordant analyses.

[redacted]

A second factor, errors in sampling procedure, is also important, and is extensively observed. Due to shortage of trained personnel, sampling is frequently and optimistically entrusted to new operators, with little understanding or judgment. Even conscientious and experienced technicians may be so preoccupied with other work that serious slips in sampling procedure result. It is a rare tour of inspection that does not reveal one or several deviations from the approved procedures. A few examples may suffice to illustrate this.

During the operation of filling an H bottle in the RC Department, it was observed that the motor circulating dry air in the dry box was not running. This went unnoticed by the operator, who made the prescribed adjustments of the motor rheostat without noticing that the motor plug was out.

In the Salvage Department, a large bottle of dark-colored liquid was being agitated, and a considerable amount of solid was observed in the solution. The amount of solid was obviously too great for the degree of stirring employed. Inspection of the card for this batch showed that it was reported as filtered. It is suspected that if the foreman's attention had not been called to the condition of this solution, a sample would have been taken without further treatment.

The technique with which EP batches are sometimes sampled seems to offer opportunity for solid material to settle out before the pipet is removed from the solution, and for loss of material on the necks and sides of sample flasks.

Instances have occurred where the neck of a weighed container has been washed with a considerable quantity of water, which was allowed to run into the container. This would of course introduce an error into the calculations of the next department which handled that material.

III. LIST OF SAMPLES TAKEN--Classified as to departmental source, material sampled, homogeneity, function of the analysis, constituents determined, disposal of source material, and pertinent remarks.

A. Primary Recovery

1. Sample of M unit wash (Uncxidized Gunk), taken when material is transferred to the Bulk Treatment storage tanks. The material contains much solid, and is analyzed by Alpha Lab. for material balance record to be charged to Bulk Treatment. It is necessary for comparison of the output of the various tracks. The estimated frequency of analysis is two per day, and the accuracy is probably about 2% low. Provision is made in the newer tracks for oxidation before transfer, which should improve the homogeneity.

2. Sample of solid material from the unit cleaning of the E end, analyzed by Alpha Lab. for record before storage. 720 and Fe are determined, and CM, MS assays are made. The samples run from 1% solid to impossible, and the estimated frequency is 7 or 8 per week.

[redacted]

[redacted]

B. Bulk Treatment

3. Sample of Unoxidized Gunk from Bulk Treatment storage, analyzed by Alpha Lab. within two hours for 720. The tolerance is 10%, and the results determine the dilution necessary in the reactor during the Bulk Treatment process. Since the precision attained is well below the tolerance, the analysis is satisfactory, in spite of the non-homogeneous nature of the Gunk. The frequency varies from 2 per shift to one in two days.

4. Sample of Oxidized Gunk from Bulk Treatment reactor, analyzed by Alpha Lab. for 720 for material balance only. It is a suspension, estimated to contain about 0.1% graphite particles. The Gunk is processed without further reference to the analysis. Results are about 2% low, and about 10 analyses are run per day.

5. Sample of weekly batch of solid residue from Sharples centrifuge in Bulk Treatment, which removes the precipitated iron. Alpha Lab. does the analysis, which is for record only. The samples are not good, but the analysis is not regarded as particularly important. 720 is determined.

6. Sample of filtered 707 decantrate (batch), analyzed weekly by Alpha Lab. for 720, for record only. Batch is made up of grab samples from discarded material, analysis may be high up to 10%.

7. Sample of solution from dissolving tank, analyzed for iron and 720 by Alpha Lab. This determines the amount of benzoate to be added for removal of the iron. This has been an experimental sample, but is now on a production basis. Sample is regarded as satisfactory.

8. Sample of benzoate precipitate removed by Alsop filter, analyzed by Alpha Lab. for 720 which is co-precipitated. The sample is regarded as satisfactory.

9. Sample of 705 decantrate (batch), analyzed weekly by Alpha Lab. for 720, for record only. Batch is made up of grab samples from discards. Analysis satisfactory.

10. Sample of Bulk Treatment solid sweepings (724 peroxide), analyzed for 720 by Alpha Lab. Estimated frequency, two per week. Material is recalcined.

11. Sample of 705-707 reprecipitation, analyzed for 720 by Alpha Lab. for record. The laboratory makes no distinction from samples (6) and (7), and the filtrate is discarded.

12. Sample of 727 (dituballate), analyzed by Alpha Lab. for 720 once or twice per week. This material may be for shipment.

13. Sample of Bulk Treatment product, analyzed by Alpha Lab. for 720, Fe, sulphate, and moisture (experimentally at present).

[redacted]

[redacted]

Analysis may show over-calcination or need for recalcination. The samples are frequently poor, and lead to a poor material balance. Frequency 3 to 5 per day.

C. Liquid Phase

14. Sample of 723 from manufacturer, analyzed by Alpha Lab. for record. 720, Fe, sulphate, and moisture (experimental at present) are determined, on sample taken as 723 container is opened (spot sample) preparatory to emptying into reactor. Frequency, 5 or 6 per week. Sample regarded as satisfactory.

no longer made. DB 4/9/49

15. Sample of hopper solid, analyzed by Alpha Lab. for 720, Cl, 751, and insolubles. Sample usually satisfactory, but sometimes contains unreacted lumps. Material goes to calciner. Frequency 2 to 7 per week.

frequency about 1 per month

16. Sample of 751 liquid suspension, analyzed by Alpha Lab. for 720. This is effluent from centrifuge, and may contain up to 1% solids. Frequency variable, about two per week.

17. Sample of product (731), analyzed by Alpha Lab. for 720, Cl, and insolubles. It usually contains small lumps of unreacted material. The purpose is to maintain the correct Cl/720 ratio, and the material is resublimed if necessary. Frequency 15 to 20 per week.

D. Sublimation

18. Sample of residue from sublimation, analyzed by Alpha Lab. for 720 and Fe, for record. A sample is taken from the residue in each boat, when it is emptied into a large can with other like residues. The samples are considered satisfactory, and the estimated frequency is 7 or 8 per week. This residue is sent back through the Bulk Treatment process.

19. Sample of line wash, which is collected in an open rectangular tank. Alpha Lab. analyses for 720, for record. Estimated solids, 1%; frequency, 1 batch per week. Material returns to Bulk Treatment for reprocessing.

20. Sample of reclaim material, in the form of a spot sample from a large can of solid. Alpha Lab. analyzes one batch sample per week for 720, Fe, Cl, and insolubles for record. The sample is considered satisfactory, and the material is returned to the retorts for resublimation.

21. Sample of sublimation product, analyzed for 720, Fe, and Cl by Alpha Lab. The samples are considered satisfactory, and the analyses are usually good. Results are for record and process information, and about 8 analyses are run per week.

[redacted]

E. Bottling [redacted]

22. Sample of bottle wash, analyzed by Alpha Lab. for 720, for record. The sample may contain some solid, and the material is sent to Bulk Treatment. One batch analyzed per week.

F. P.S.W.

23. Sample of E Pocket washing and leaching, for individual boxes. Analyzed by Beta Lab. for 720, for record. Sample usually satisfactory, and variation attributed to personal sampling errors. Frequency estimated, 15 to 30 batches daily. ?

1/13 x individuals done All Batches sample taken

24. Sample of production batch, E Pocket washing and leaching (739). Analyzed by Beta Lab. for 720, assayed by MS, for record. Samples usually good, but some solid is occasionally found. Frequency, 4 to 6 batches daily. Samples from 9206 are very good.

25. Sample of "D" batch, which is a composite of production batches and individuals, analyzed by Beta Lab.

G. R.O.

26. Sample of aqueous solution from ether extraction, prepared by Beta Lab. for CM assay (720). Material is extracted further if necessary. Samples seem satisfactory, but variation in CM results possibly indicate lack of sample homogeneity. Frequency, 4 to 10 batches per day.

27. Sample of calcined oxide (R.O. batch numbers), R.O. product. Analyzed by Beta Lab. for record (720). Some batches are contaminated with iron. Frequency 10 batches daily.

H. R.C.

28. Samples of wash in RC. 964 is soluble, and is line wash. 924 may contain unconverted oxide from XRO in 705. Beta Lab. analyzes about 2 batches per day for 720, for record. The homogeneity is usually considered good.

29. Sample of bottle charge (solid) for Beta Tracks, by Beta Lab. for record. Distribution of iron in the source material is often non-homogeneous, and samples are therefore taken from both top and bottom of the tube. Frequency about 10 to 15 batches daily.

I. R.R.

30. Sample of evaporated machine-wash (E.P., 738, Beta Gunk), analyzed by 9204-1 lab. for 720 by rapid method, with a frequency of about 80 per week. Analysis is for control of extraction process in XRO. Material is sent to XRO. Duplicate

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samples are sent to Henrickson for check analyses by slower but more precise methods. These are used for record. Nitrates are also run by 9204 laboratory on some samples. Sample contains solid.

31. Sample of bottle wash (XW), analyzed by rapid method in 9204-1 for 720, and used for extraction process information. Samples contain solids, and about 30 per week are analyzed. Material goes to XRO for processing.

32. Sample of distillate from evaporator, analyzed by 9204-1 laboratory for acid content and chloride before re-use in washing machines. About 3 samples are run each week.

J. X.R.O.

33. Sample of aqueous solution remaining after ether extraction, prepared for CM assay and analyzed in Beta Lab. About 10 to 15 batches are handled per day, and discordant results of CM suggest that homogeneity may be questionable. The results determine if further extraction is necessary.

34. Sample of calcined oxide (XRO batch numbers), analyzed by Beta Lab. for 720 by ignition, for record charged to RC. Iron contamination is noted, but batches seem homogeneous. About 10 to 15 batches are analyzed daily.

K. R.S.W.

35. Sample of Q washings, prepared for CM assay by Products Lab. before sending to salvage. Sample considered satisfactory. Detailed information restricted.

36. Sample of unresolved material washing, analyzed for 720 by Products Lab. and prepared for CM assay. Considered homogeneous. Detailed information restricted.

37. Sample of R washings (solution) from carbon ash leach, ana. by Products Lab. and prepared for MS assay. Considered homogeneous. Details restricted.

38. Sample of 705 reprecipitation filtrate, analyzed by Products Lab. Details restricted. Considered homogeneous.

L. Product

39. Sample of oxide product, analyzed by Products Lab. and prepared for MS assay. Details restricted.

40. Sample of final product, analyzed for specifications by Products Lab. Spectrographic analysis included. Considered homogeneous, further details restricted.

[redacted]

[redacted]

M. Salvage

Because of the difficult nature of much of the material handled by the Salvage Department, incoming material is not usually sampled and analyzed. Salvage is credited with material on the basis of analyses done upon samples taken after the salvage process is completed.

It is estimated that about 5 to 7 analyses per day are done for salvage, and the samples are regarded as often unsatisfactory, possibly due to lack of care and discretion in taking them, or to inadequate supervision of the sampling in the past.

The following list shows the sources of material processed by salvage, with notations as to their relative richness, and destination after processing.

1. PSW:-stripping solution--medium rich--goes to RO

2. RO-XRO:-rags and paper--poor--goes to RO or XRO

3. RO-XRO:-705 filtrate--rich--goes to RO or XRO

4. RO-XRO:-gloves--goes to RO or XRO

5. RC:-707 scrub solution--very poor--goes to XRO

6. RC:-751 trap solution--very poor--goes to XRO

7. RC:-rags, paper, etc.--poor--goes to XRO

8. RR:-filter pads--rich--goes to XRO

9. RR:-M carbons--rich--goes to XRO

10. RR:-rags, paper, etc.--goes to XRO

11. Beta Lab.:-(CM)--concentrate--rich--goes to RO

12. Beta Lab.:-combined 707-705 filtrates from RO, XRO, RC, and Beta

13. Beta Lab.:-oxalate filtrate concentrate--goes to storage

14. Beta Lab.:-paper--very poor--goes to RO

15. RSW:-unresolved carbons, ashed, leached--medium rich--to XRO or storage-Products Lab. analyzes these

16. Residues for storage--from various sources--CM assay.

17. Sample of FR (filter residue ?), insoluble residues from PSW and XRO. These will eventually go to Henrickson. Considered to be difficult samples. Analyzed by Products Lab. at present.

[redacted]

[redacted]

IV. GENERAL CONCLUSIONS

A. Approximately 15 analyses out of the total number of determinations listed are actually essential to the process. The rest are used to determine the material balance at various stages of the process.

B. Approximately 15 - 18 samples are considered not to be consistently homogeneous and representative.

C. Personal errors in sampling were found to be quite prevalent, probably due to lack of responsibility and supervision.

V. RECOMMENDATIONS

A sampling laboratory should be set up, staffed with competent and mature personnel, whose authority is commensurate with the responsibility involved.

Equipment, such as grinders, mixers, riffles, sampling tubes, and agitators should be on hand so that all types of materials can be quickly sampled.

Sampling personnel should not be connected with the departments in which the samples are taken, and should be selected for proper temperament, particularly trustworthiness in attention to detail. They should also have some training in sampling procedures.

Material to be sampled should be inspected at the source by a representative of the Sampling Department, who will take the sample if the material is homogeneous. In cases where it is more appropriate for the operator to take the sample, such as in dry box operations, the sampling representative should supervise the taking of the samples by the operator, and take charge of the resulting samples.

Material which is not homogeneous, and cannot be readily rendered so at the source, may then be transported to the sampling laboratory for suitable preparation and sampling.

In special cases, such as experimental material, where the tolerance of error is high and sampling is difficult, it should be possible to sample by the most feasible means, and designate the sample as "doubtful". This designation should be retained on the record of that sample and its analysis.

C. L. Smith

11-21-44

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